National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 999b

Potassium Chloride

(Primary Chemical)

This Standard Reference Material (SRM) is intended for use as an analytical standard of known potassium (K) and chloride (Cl^-) content. This lot of potassium chloride (KCl) was prepared to ensure a material of high purity and homogeneity and has been assayed after ignition at 500 °C. A unit of SRM 999b consists of a single glass bottle containing 30 g of the material.

Table 1 lists the certified values for this SRM, expressed as mass fractions, *w*, of KCl, K, and Cl^-. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1].

Table 1. Certified Values[(a)] for SRM 999b Potassium Chloride

w_{KCl}	99.977 %	±	0.014 %
w_{Cl}	47.5519 %	±	0.0046 %
w_K	52.4379 %	±	0.0084 %

[(a)] Each result is expressed as the certified value ± the expanded uncertainty, *U*, calculated as $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of inherent sources of uncertainty of the assay techniques and applicable corrections for interfering trace elements. The value of the coverage factor, *k*, is 1.96, which corresponds to approximately 95 % confidence based on >1000 effective degrees of freedom.

Expiration of Certificate: The certification of this SRM is valid until **01 August 2015**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. The certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of Certification: NIST will monitor representative samples from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coulometric and gravimetric analyses were performed in the NIST Analytical Chemistry Division by K.W. Pratt and T.W. Vetter, respectively. Trace bromine (Br) determination by X-ray fluorescence was performed in the NIST Analytical Chemistry Division by J.R. Sieber. Additional trace element analyses by glow-discharge mass spectrometry were performed by a commercial laboratory.

Coordination of the technical measurements leading to the certification of SRM 999b was provided by K.W. Pratt of the NIST Analytical Chemistry Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

The support aspects involved in the preparation of this SRM were coordinated through the NIST Measurement Services Division.

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Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 March 2006

Robert L. Watters, Jr., Chief
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Table 2 lists reference values for the mass fractions of Br and excess alkalinity, expressed as potassium hydroxide (KOH) referred to the solid, dried SRM 999b. The alkalinity was obtained from pH measurements in a carbon dioxide-free 1.0 mol/L solution of SRM 999b. The expanded uncertainties are calculated using $k = 2$. Reference values are a best estimate of the true value provided by NIST where all known or suspected sources of bias have not been fully investigated by NIST [1].

Table 2. Reference Values for SRM 999b

Element or Property	Mass Fraction (µg/g)	Expanded Uncertainty (µg/g)
Br	130	45
Alkalinity (as KOH)	1.19	0.27

Table 3 lists information values for SRM 999b. No other elements were detected at a mass fraction greater than 1 µg/g. Information values are non-certified values that may be of interest and use to the SRM user, but insufficient information is available to provide an uncertainty associated with the value [1].

Table 3. Information Values for SRM 999b

Element	Mass Fraction (µg/g)
Na	35
Rb	2.6
Si	1.8

NOTICE AND WARNINGS TO USERS

The certified value for w_{KCl} is obtained from a weighted combination of the results of independent coulometric analyses, corrected for the potassium bromide (KBr), rubidium chloride (RbCl), and sodium chloride (NaCl) impurities; and gravimetric analyses, corrected for the KBr impurity.

The certified value for w_{Cl} is obtained from the coulometric analyses, corrected for interfering bromide.

The certified value for w_K is obtained from an equally-weighted combination of w_K obtained directly from the gravimetric analyses and the indirect w_K, which is calculated from the coulometric w_{KCl} and the additional K in the KBr impurity.

The corrections for bromide, sodium, and rubidium were obtained from the trace element determinations and the appropriate gravimetric factors. A portion of the K is present in SRM 999b as KBr, and a portion of the chloride is present as NaCl and RbCl. Hence, the sum of the certified values for w_K and w_{Cl} does not equal the certified value for w_{KCl}.

It is the responsibility of the user to ascertain which species may interfere with the application of this SRM and to apply any necessary corrections that affect the given application.

INSTRUCTIONS FOR USE

Drying Instructions: Dry for 4 h at 500 °C in platinum or fused silica (borosilicate glass is unsatisfactory) vessels. After the SRM has been dried, store it in a desiccator over anhydrous magnesium perchlorate and gently crush any lumps of KCl present before using.

Stability and Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and light.

Homogeneity: This SRM is homogeneous within the uncertainty limits for the nominal sample mass, 250 mg, used for the coulometric assays. Samples less than 250 mg are not recommended in order to avoid possible heterogeneity with smaller sample sizes.

Source of Material: The KCl used for this SRM was obtained from a commercial supplier. The material was examined for compliance with the specification for reagent grade KCl as specified by the American Chemical Society [3]. The material was found to meet or exceed the minimum requirements in every respect.

Assay Techniques: The coulometric assay value was obtained by automated titration [4] with coulometrically generated Ag^+ using potentiometric detection of the endpoint. The gravimetric assay value was obtained by ion-exchange separation of the K fraction and conversion to potassium sulfate (K_2SO_4), including corrections for instrumentally-determined K not collected with the K fraction and for trace contaminants in the K_2SO_4 (procedure based on [5]).

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at http://ts.nist.gov/ts/htdocs/230/232/SP_PUBLICATIONS/documents/sp260-136.pdf

[2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, lst ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs.

[3] *Reagent Chemicals*, 9th ed., American Chemical Society: Washington, DC (1999).

[4] Pratt, K.W.; *Automated, High-Precision Coulometric Titrimetry Part I. Engineering and Implementation*; Anal. Chim. Acta, Vol. 289, pp. 125–134 (1994).

[5] Moody, J.R.; Vetter, T.W.; *Development of the Ion Exchange-Gravimetric Method for Sodium in Serum as a Definitive Method*; J. Res. Natl. Inst. Stand. Technol., Vol. 101, pp. 155–164 (1996); available at http://nvl.nist.gov/pub/nistpubs/jres/101/2/j2mood.pdf.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm